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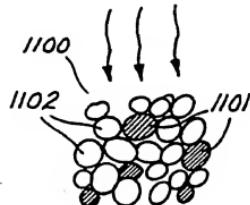
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(54) Title: PRODUCING PARTS BY COMPOUND FORMATION OF PRECURSOR POWDERS



## (57) Abstract

A method and apparatus for selectively sintering a layer of powder to produce a part comprising a plurality of sintered layers. The apparatus includes a computer controlling a laser to direct the laser energy onto the powder to produce a sintered mass. The computer either determines or is programmed with the boundaries of the desired cross-sectional regions of the part. For each cross-section, the aim of the laser beam is scanned over a layer of powder and the beam is switched on to sinter only the powder within the boundaries of the cross-section. Powder is applied and successive layers sintered until a completed part is formed. Preferably, the powder comprises a plurality of materials having different dissociation or bonding temperatures. The powder preferably comprises blended or coated materials, including precursor materials which are formed into a mass at the irradiated locations, and which either react due to the laser thermal energy or in a later heat treatment to form a compound with properties different than the precursors. Examples are disclosed wherein a compound is formed which has a significantly higher melting point than that of one or more of the precursor powders.

## PRODUCING PARTS BY COMPOUND FORMATION OF PRECURSOR POWDERS

\* \* \* \* \*

5        This invention relates to a method and apparatus which uses a directed energy beam to selectively sinter a powder to produce a part. In particular, this invention relates to a computer aided laser apparatus which sequentially sinters a plurality of powder layers to build the desired part in a 10 layer-by-layer fashion. The present application is particularly directed towards a powder comprising a plurality of materials where the powder has more than one bonding or dissociation temperature.

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Background of the Invention

The economies associated with conventional part production methods are generally related directly to the quantity of parts 20 to be produced and the desired material characteristics of the finished parts. For example, large scale manufacture casting and extrusion techniques are often cost effective, but these production methods are generally unacceptable for small quantities, i.e. replacement parts or prototype production. 25 Many such conventional part production methods require expensive part specific tooling. Even powder metallurgy requires a die for shaping the powder, making powder metallurgy unattractive as a method for producing a small number of parts.

30        Where only a small number of parts are desired, conventional production methods involving a subtractive machining method are usually used to produce the desired part. In such subtractive methods, material is cut away from a starting block of material to produce a more complex shape. 35 Examples of subtractive machine tool methods include: milling,

drilling, grinding, lathe cutting, flame cutting, electric discharge machine, etc. While such conventional machine tool subtractive methods are usually effective in producing the desired part, they are deficient in many respects.

5

First, such conventional machine tool subtractive methods produce a large amount of waste material for disposal. Further, such machine tool methods usually involve a large initial expense for setting up the proper machining protocol 10 and tools. As such, the set-up time is not only expensive, but relies a great deal on human judgment and expertise. These problems are, of course, exacerbated when only a small number of parts are to be produced.

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Another difficulty associated with such conventional machining techniques involves tool wear - which not only involves the cost of replacement, but also reduces machining accuracy as the tool wears. Another limit on the accuracy and tolerance of any part produced by conventional machining 20 techniques is the tolerance limits inherent in the particular machine tool. For example, in a conventional milling machine or lathe, the lead screws and ways are manufactured to a certain tolerance, which limits the tolerances obtainable in manufacturing a part on the machine tool. Of course, the 25 tolerances attainable are reduced with age of the machine tool.

30

The final difficulty associated with such conventional machine tool subtractive processes is the difficulty or impossibility of making many part configurations. That is, conventional machining methods are usually best suited for producing symmetrical parts and parts where only the exterior 35 part is machined. However, where a desired part is unusual in shape or has internal features, the machining becomes more difficult and quite often, the part must be divided into segments for production. In many cases, a particular part

configuration is not possible because of the limitations imposed upon the tool placement on the part. Thus, the size and configuration of the cutting tool do not permit access of the tool to produce the desired configuration.

5

There are other machining processes which are additive, for example, plating, cladding, and some welding processes are additive in that material is added to a starting substrate. In recent years, other additive-type machining methods have been 10 developed which use a laser beam to coat or deposit material on a starting article. Examples include U.S. Patent Nos. 4,117,302; 4,474,861; 4,300,474; and 4,323,756. These recent uses of lasers have been primarily limited to adding a coating to a previously machined article. Often such laser coating 15 methods have been employed to achieve certain metallurgic properties obtainable only by such coating methods. Typically, in such laser coating methods the starting article is rotated and the laser directed at a fixed location with the coating material sprayed onto the article so that the laser will melt 20 the coating onto the article.

U.S. Patents No. 4,944,817, No. 4,863,538, No. 4,938,816, and PCT publication WO 88/02677 published 21 April 1988, all of which are incorporated herein by this reference, describe a 25 method of producing complex parts directly from a CAD data base which is not subject to the above-described limitations of the various subtractive and additive methods; this new method will be referred to as "selective beam sintering" or "selective laser sintering". The selective laser sintering process is 30 particularly advantageous in forming prototypes for parts which may subsequently be mass produced, for example by investment casting, or by the use of tooling.

It is desirable to utilize the selective beam sintering 35 process not only to produce prototype models of subsequently

mass produced parts, but also to produce parts suitable for actual use in a machine. Such usable parts may also be prototypes, useful for testing the structural design of the part and the system into which it is to be incorporated; in 5 addition, it is desirable to produce such parts which are capable of serving in the end equipment product.

The material of parts which are to be actually used must be of sufficient strength and integrity to meet the mechanical and 10 temperature stress requirements of its application. Accordingly, the formation of intermetallic and ceramic (including glass) parts by selective beam sintering is desirable. However, many desirable metallic and ceramic 15 powders have sintering or melting temperatures which are sufficiently high that selective sintering or melting by a directed energy beam, such as a laser, is not easily achievable at this time.

It is therefore an object of this invention to provide a 20 method of producing a part by the application energy to selected portions of a multiple material powder, where the properties of the material of the part produced differs from those of the materials in the powder.

25 It is a further object of this invention to provide such a method where the melting temperature of the produced part is higher than the temperature to which the directed beam subjects the powder.

30 It is a further object of this invention to provide such a method where the material used is a metal or ceramic.

35 It is a further object of this invention to provide such a method where the material of the part is a chemical compound of the constituents of the powder.

It is a further object of this invention to provide such a method which includes post-processing to cause the chemical reaction.

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Other objects and advantages of the invention will be apparent to those of ordinary skill in the art having reference to the following specification, together with the drawings.

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Summary of the Invention

The problems outlined above are in large measure solved by 5 the method and apparatus of the present invention. The present invention includes a directed energy beam - such as a laser - and is adaptable to produce almost any three dimensional part. The method of the present invention is an additive process, with the powder being dispensed into a target area where the 10 laser selectively sinters the powder to produce a sintered layer. The invention is a layer-wise process in which the layers are joined together until the completed part is formed. The method of the present invention is not limited to a particular type of powder, but rather is adaptable to plastic, 15 metal, polymer, ceramic, wax, semiconductor or amorphous powders, or composite material powders.

Broadly speaking, the apparatus includes a laser or other directed energy source which is selectable for emitting a beam 20 in a target area where the part is produced. A powder dispenser system deposits powder into the target area. A laser control mechanism operates to move the aim of the laser beam and modulates the laser to selectively sinter a layer of powder dispensed into the target area. The control mechanism operates 25 to selectively sinter only the powder disposed within defined boundaries to produce the desired layer of the part. The control mechanism operates the laser to selectively sinter sequential layers of powder, producing a completed part comprising a plurality of layers sintered together. The defined boundaries of each layer correspond to respective 30 cross-sectional regions of the part. Preferably, the control mechanism includes a computer - e.g. a CAD/CAM system - to determine the defined boundaries for each layer. That is, given the overall dimensions and configuration of the part, the 35 computer determines the defined boundaries for each layer and

operates the laser control mechanism in accordance with the defined boundaries. Alternatively, the computer can be initially programmed with the defined boundaries of each layer.

5        In a preferred form, the laser control mechanism includes a mechanism for directing the laser beam in the target area and a mechanism for modulating the laser beam on and off to selectively sinter the powder in the target area. In one embodiment, the directing mechanism operates to move the aim of  
10      the laser beam in a continuous raster scan of target area. The modulating mechanism turns the laser beam on and off so that the powder is sintered only when the aim of the laser beam is within the defined boundaries for the particular layer. Alternatively, the directing mechanism aims the laser beam only  
15      within the defined boundaries for the particular layer so that the laser beam can be left on continuously to sinter the powder within the defined boundaries for the particular layer.

In a preferred embodiment, the directing mechanism moves  
20      the laser beam in a repetitive raster scan of the target area using a pair of mirrors driven by galvanometers. The first mirror reflects the laser beam to the second mirror which reflects the beam into the target area. Shifting movement of the first mirror by its galvanometer shifts the laser beam  
25      generally in one direction in the target area. Similarly, shifting movement of the second mirror by its galvanometer shifts the laser beam in the target area in a second direction. Preferably, the mirrors are oriented relative to each other so that the first and second directions are generally  
30      perpendicular to each other. Such an arrangement allows for many different types of scanning patterns of the laser beam in the target area, including the raster scan pattern of the preferred embodiment of the present invention.

35        The method of part production of the present invention

includes the steps of depositing a first portion of powder onto a target surface, scanning the aim of a directed energy beam (preferably a laser) over the target surface, and sintering a first layer of the first powder portion on the target surface.

5      The first layer corresponds to a first cross-sectional region of the part. The powder is sintered by operating the directed energy source when the aim of the beam is within the boundaries defining the first layers. A second portion of powder is deposited onto the first sintered layer and the aim of the

10     laser beam scanned over the first sintered layer. A second layer of the second powdered portion is sintered by operating the directed energy source when the aim of the beam is within the boundaries defining the second layer. Sintering of the second layer also joins the first and second layers into a cohesive mass. Successive portions of powder are deposited onto the previously sintered layers, each layer being sintered in turn. In one embodiment, the powder is deposited continuously into the target.

20     In a preferred embodiment, the laser beam is modulated on and off during the raster scan so that the powder is sintered when the aim of the beam is directed within the boundaries of the particular layer. Preferably, the laser is controlled by a computer; the computer may include a CAD/CAM system, where

25     the computer is given the overall dimensions and configuration of the part to be made and the computer determines the boundaries of each cross-sectional region of the part. Using the determined boundaries, the computer controls the sintering of each layer corresponding to the cross-sectional regions of the part. In an alternative embodiment, the computer is simply programmed with the boundaries of each cross-sectional region of the part.

30     Additionally, another embodiment of the present invention includes a powder comprising a plurality of materials where the

plurality of materials have more than one dissociation temperature. In still another embodiment of the present invention, the powder comprises a plurality of materials where the plurality of materials have more than one bonding 5 temperature.

As used throughout this document, bonding temperature includes but is not limited to, melting temperature, softening temperature and bonding temperature.

10

In all preferred embodiments of the present invention, the plurality of materials comprise at least one first material blended with at least one second material or at least one first material coated with at least one second material.

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As can be appreciated from the above general description, the method and apparatus of the present invention solves many of the problems associated with known part production methods.

20

First, the present invention is well suited for prototype part production or replacement part production of limited quantities. Further, the method and apparatus hereof are capable of making parts of complex configurations unobtainable by conventional production methods. Further, the present invention eliminates tool wear and machine design as limiting factors on the tolerances obtainable in producing the part. Finally, with the apparatus of the present invention incorporated into a CAD/CAM environment, a large number of replacement parts can be programmed into the computer and can be easily produced with little set-up or human intervention.

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The method according to the invention may also be applied to produce a part of a material having one or properties different from those of the constituent materials in the multiple material powder. The application of thermal energy 30 to selected locations of particular multiple material powders

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enables a chemical reaction, and the formation of a stable or metastable chemical compound, at the locations of the powder exposed to the energy. The reaction may occur during the application of the directed energy; alternatively, the directed  
5 energy may melt or sinter the materials into a mass at the selected locations, with subsequent heat treatment causing the chemical reaction. The compound may have important properties which are different from the properties of any one of the powder constituents, such as a much higher melting point. As  
10 a result, a part may be formed from a material which is otherwise not conducive to selective laser or beam sintering.

Brief Description of the Drawings

FIGURE 1 is a schematic representation of the apparatus of  
5 the present invention;

FIGURE 2 is a schematic showing a portion of the layered  
build up of a part produced in accordance with the present  
invention, and illustrating the raster scan pattern of the  
10 laser beam in the target area;

FIGURE 3 is a block diagram depicting the interface  
hardware between the computer, laser and galvanometers of the  
present invention;

15 FIGURE 4 is a perspective view of an example part produced  
in accordance with the present invention;

FIGURE 5 is a sectional view with parts broken away and in  
20 phantom, of the part illustrated in FIGURE 4;

FIGURE 6 is a flow chart of the data metering program in  
accordance with the present invention;

25 FIGURE 7 is a sectional view taken along line 7-7 of FIGURE  
4;

FIGURE 8 illustrates in diagram form the correlation  
between a single sweep of the laser across the layer of FIGURE  
30 7 and the control signals of the present invention;

FIGURE 9 illustrates a blend of materials in a powder;

FIGURE 10 illustrates materials coated in a powder;

12

FIGURE 11 illustrates a portion of a sintering cycle on a blend of materials as presently understood.

FIGURE 12 illustrates two materials deposited prior to  
5 sintering.

FIGURES 13 and 14 are phase diagrams of binary metal systems usable in an alternative embodiment of the invention.

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Detailed Description of the Preferred Embodiments

Turning now to the drawings, FIGURE 1 broadly illustrates the apparatus 10 in accordance with the present invention. Broadly speaking, the apparatus 10 includes a laser 12, powder dispenser 14, and laser control means 16. In more detail, the powder dispenser 14 includes a hopper 20 for receiving the powder 22 and having an outlet 24. The outlet 24 is oriented for dispensing the powder to a target area 26, which in FIGURE 1 is generally defined by the confinement structure 28. Of course, many alternatives exist for dispensing the powder 22.

The components of the laser 12 are shown somewhat schematically in FIGURE 1 and include a laser head 30, a safety shutter 32, and a front mirror assembly 34. The type of laser used is dependent upon many factors, and in particular upon the type of powder 22 that is to be sintered. In the embodiment of FIGURE 1, a Nd:YAG laser (Lasermetrics 9500Q) was used which can operate in a continuous or pulsed mode with a hundred-watt maximum outlet power in the continuous mode. The laser beam output of the laser 12 has a wavelength of approximately 1060 nm, which is near infrared. The laser 12 illustrated in FIGURE 1 includes an internal pulse rate generator with a selectable range of about one kiloHertz to forty kiloHertz, and an approximately six nanosecond pulse duration. In either the pulsed or continuous mode, the laser 12 can be modulated on or off to selectively produce a laser beam which travels generally along the path shown by the arrows in FIGURE 1.

30

To focus the laser beam, a diverging lens 36 and converging lens 38 are disposed along the path of travel of the laser beam as shown in FIGURE 1. Using just the converging lens 38, the location of the true focal point is not easily controlled by varying the distance between the converging lens 38 and the

laser 12. The diverging lens 36 placed between the laser 12 and converging lens 38 creates a virtual focal point between the diverging lens 36 and the laser 12. Varying the distance between the converging lens 38 and the virtual focal point, 5 allows control of the true focal point along the laser beam path of travel on the side of the converging lens 38 remote from the laser 12. In recent years there have been many advances in the field of optics, and it is recognized that many alternatives are available to efficiently focus the laser beam 10 at a known location.

In more detail, the laser control means 16 includes computer 40 and scanning system 42. In a preferred embodiment, the computer 40 includes a microprocessor for controlling the 15 laser 12 and a CAD/CAM system for generating the data. In the embodiment illustrated in FIGURE 1, a personal computer is used (Commodore 64) whose primary attributes include an accessible interface port and a flag line which generates a nonmaskable interrupt.

20 As shown in FIGURE 1, the scanning system 42 includes a prism 44 for redirecting the path of travel of the laser beam. Of course, physical layout of the apparatus 10 is the primary consideration in determining whether a prism 44, or a plurality 25 of prisms 44, are needed to manipulate the path of travel of the laser beam. The scanning system 42 also includes a pair of mirrors 46, 47 driven by respective galvanometers 48, 49. The galvanometers 48, 49 coupled to their respective mirrors 46, 47 to selectively orientate the mirrors 46, 47. The galvanometers 30 46, 47 are mounted perpendicular to each other such that the mirrors 46, 47 are mounted nominally at a right angle to each other. A function generator driver 50 controls the movement of the galvanometer 48 (galvanometer 49 is slaved to the movement of galvanometer 48) so that the aim of the laser beam (represented by the arrows in FIGURE 1) can be controlled in the 35

target area 26. The driver 50 is operatively coupled to the computer 40 as shown in FIGURE 1. It will be appreciated that alternative scanning methods are available for use as the scanning system 42, including acusto-optic scanners, rotating 5 polygon mirrors, and resonant mirror scanners.

Turning to FIGURE 2 of the drawing, a portion of a part 52 is schematically illustrated and shows four layers 54-57. The aim of the laser beam, labeled 64 in FIGURE 2, is directed in 10 a raster scan pattern as at 66. As used herein, "aim" is used as a neutral term indicating direction, but does not imply the modulation state of the laser 12. For convenience, the axis 68 is considered the fast scan axis, while the axis 70 is referred to as the slow scan axis. Axis 72 is the direction of part 15 build-up.

Turning to FIGURES 9 and 10, powders, comprising a plurality of materials by which parts may be made using the present invention, are illustrated. For simplicity, only two 20 materials are shown in the illustrations. However, as will be apparent to one skilled in the art, a plurality of materials may comprise the powder of the present invention.

FIGURE 9 illustrates a blend of first material 901 and 25 second material 902. The materials are combined in a blend through conventional blending processes. FIGURE 10 illustrates material 1002 coated with material 1001. Material 1002 is coated using conventional coating processes.

30 As will be further apparent to one skilled in the art, coated materials, as shown in FIGURE 10, may be blended to produce a desired mix of materials.

Turning to FIGURE 11, a portion of a sintering cycle, as 35 presently understood, is illustrated. FIGURE 11a illustrates

a blend of materials prior to the application of energy able to produce sintering. Preferably, the materials comprising powder mass 1100 have more than one bonding or dissociation temperature. FIGURE 11b illustrates powder 1100 during 5 application of energy sufficient to promote sintering. FIGURE 11b illustrates material 1101 having a lower bonding or dissociation temperature than material 1102. In a preferred embodiment, the low temperature phase material 1101 infiltrates powder mass 1100 in the area surrounding each particle of 10 material 1101. Additional powder components could also be added to the blend to promote infiltration. Similarly a gas phase can be used to promote infiltration and the sintering process. The gas phase may be either inert or active, preferably to either displace an undesired gas or introduce a 15 desired gas. FIGURE 11c illustrates a potential mechanism through which effects, including but not limited to, capillarity effects, allow material 1101 to infiltrate the powder mass 1100. FIGURE 11d shows the part following sintering in the present invention.

20 Because a material having a bonding or dissociation temperature higher than the temperature to be obtained during the sintering process may be selected, the higher bonding or dissociation temperature material need not sinter but may 25 retain its original structure. In particular, for crystalline material this enables control of epitaxial growth in the selective beam sintering process of the present invention. For example, if the higher bonding or dissociation temperature material is positioned in a particular structure that may, 30 preferably, result in epitaxial growth from the preceding layer, only bonding or dissociating the lower bonding or dissociation temperature material enables the higher temperature material to retain its structure.

35 The choice of materials selected for the powder allows for

a broad range of resulting sintered material. For example, a conducting material is preferably coated with an insulating polymer material to produce a powder. The powder is then distributed in the target area. The material is preferably 5 sintered and the insulator may be removed later through a conventional process, including but not limited to a chemical method, resulting in a conductive, sintered product.

By way of further illustration, extremely hard materials 10 may be produced using the present invention. For example, tungsten carbide/cobalt tools which, because of their extreme hardness are difficult to form or sharpen may be produced by coating tungsten carbide material with cobalt to produce a powder or by blending tungsten carbide and cobalt to produce a 15 powder. During sintering, the cobalt preferably melts under the applied energy beam causing local infiltration of the tungsten carbide. The part that is manufactured is ready to be put into service preferably after a secondary process including, but not limited to, annealing.

20 By way of further illustration, copper and tin may be combined in a powder. Tin, having a lower melting temperature than copper, will melt and infiltrate the copper during sintering.

25 Secondary processing may also be applied to parts produced using the present invention. For example, where tin is allowed to melt and infiltrate copper during sintering, post process annealing will dissolve the tin into the copper in the solid 30 state creating bronze with minimal volume change or distortion.

By way of further illustration, metal, including but not limited to, iron or steel, may be coated with poly(methyl methacrylate) (PMMA) polymer to form a powder. Sintering 35 enables the PMMA to flow and bind the metal. Post process

annealing will dissociate the PMMA and sinter the metal thus producing a final part.

Ceramic materials may be processed in this fashion as well.

5     For example, a mixture of fluorophosphate glass powders with alumina powders will result in the glass softening and infiltrating the alumina during the sintering process. In another example, aluminum silicate, silica, or other ceramic powder can be coated with a polymer by a variety of methods,

10    including spray drying and solvent coating. A surface active agent may be used to pretreat the ceramic powder prior to coating. This agent may be based on organosilane chemistry or other chemistries known to promote the wettability of the ceramic by the polymer and the adhesion of ceramic to polymer.

15    Any polymer, either thermoplastic or thermoset, which can be coated on the ceramic, can be used as a binder. Typical materials include PMMA, polystyrene, various epoxy formulations, and phenolics.

20    Any combination of materials, including but not limited to, metals, ceramics and polymers enables production of parts in accordance with the present invention wherein at least one material in the powder has a low bonding or dissociation temperature relative to the other materials in the powder.

25    In another preferred embodiment of the present invention, the temperature of the powder mass may be increased using conventional heating means allowing the energy beam to merely supply a small increase of energy to produce bonding or dissociation of one of the elemental materials of the powder.

30    Materials comprising the powder may be chosen for each material's selective absorption of energy from a laser beam (represented by the arrows in FIGURES 11a and 11b). In the preferred embodiment shown in FIGURE 11, material 1101 may be

chosen to absorb the wavelength of the applied beam energy represented by the arrows while elemental material 1102 absorbs less energy thereby enabling elemental material 1101 to bond or dissociate prior to the bonding or dissociation of elemental 5 material 1102. This absorption of energy can be achieved by either material or laser beam wavelength selection, or both, in a plurality of combinations.

According to another alternative embodiment of the 10 invention, the application of the directed energy beam to selected portions of the powder can be used to enable a chemical reaction of the constituent materials within the powder. In this embodiment, the powder includes multiple precursors of the compound to be formed, for example a mixture 15 of elemental or alloyed powders. The energy of the directed energy beam in the selective laser sintering process causes the formation of a mass at the locations of each powder layer which are exposed to the directed beam. The part is built up by the layerwise process of placing powder at the target surface and 20 selective laser sintering or binding selected portions of the layer, forming a part in layerwise fashion as described hereinabove. Upon completion of the this process, the portions of the powder not exposed to the directed energy beam are removed, as noted hereinabove.

25 Either during the laser exposure, or in a post-selective laser sintering anneal, the precursors at the irradiated locations interdiffuse, resulting in a new phase of the material based on a chemical reaction. This new phase 30 preferably has significantly different properties than any of the precursor materials; examples of these different properties include physical, mechanical, tribological, or electrical properties.

35 For example, a part which is to be subjected to high

temperatures in its intended use preferably is formed of a high melting point material. High melting point powders are not suitable for use in the selective laser sintering process, however, since the laser power will generally be insufficient

5 to melt or sinter the powder into a mass. According to this embodiment of the invention, the high melting point material of which the part is to be produced is a compound of precursors, one of which has a relatively low melting point.

10 By way of example, a compound  $A_mB_n$  may be selected which has a high melting point, where elemental constituents A and B can exist in mixed powder form. It should be noted that this method is also applicable for systems where some solid solubility of constituents in the compound exists, in which case m and n are not integers. Certain considerations are important in selecting the compound  $A_mB_n$ . Firstly, there must be a significant difference in the melting points of at least two of the precursor elements A and B such that the power capability of the laser (or other directed energy beam) is able

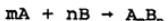
15 20 to melt the material having the lower one of the melting points while not melting the material with the higher melting point. It should be noted that the laser need not supply all of the thermal power required to melt the low melting point powder; the ambient temperature of the powder may be maintained near the lower melting point, so that the laser power is sufficient

25 30 to melt the lower melting point precursor in the irradiated locations, but not in the non-irradiated locations. Secondly, a compound  $A_mB_n$  of the precursors which has the desired properties (e.g., high melting point) must exist. Thirdly, in order that the chemical reaction can be performed in a time efficient manner, it is desirable that the reaction rate-limiting precursor species have high chemical diffusivity in the other precursor species and in the compound. Fourthly, it is desirable that the volume fraction of the low melting point

35 precursor of the total volume be significant, preferably on the

order of 25 to 75 percent, so that the molten precursor may fully infiltrate the non-melting precursor, resulting in high density parts.

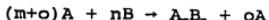
5        Generally, a single phase binary compound  $A_mB_n$  according to this embodiment of the invention results from the following reaction:



10

Alternatively, an excess amount of one of the constituent precursors may be used to produce a two phase product. For example, where an excess amount of precursor A is used, the following reaction occurs:

15



Where two compounds exist for a system, proper control of the precursor amounts in the powder can produce a product with two 20 compound phases, according to the following reaction:



FIGURE 13 is a phase diagram for a first preferred example 25 of a binary compound which can be formed according to this embodiment of the invention. The phase diagram of FIGURE 13 illustrates the melting points of various mixtures of nickel and tin. It should be noted that the melting points of elemental nickel and tin differ quite widely, with the melting 30 point of elemental tin being 231.8°C. It should also be noted that intermetallic compounds of nickel and tin exist which have high melting points; for example, the melting point of  $Ni_3Sn_2$  is on the order of 1260°C.

35

According to this embodiment of the invention, the powder

which is to undergo selective laser sintering includes a mixture of nickel and tin, with the mixture preferably having approximately 40% nickel and 60% tin (atomic percent). A part is formed by layerwise selective laser sintering, in the manner described hereinabove. Preferably, a conventional laser, such as a CO<sub>2</sub> or YAG laser, having a power on the order of 25 watts or greater, provides the directed energy beam. During selective laser sintering, the powder is preferably in a non-oxidizing atmosphere such as nitrogen; alternatively, oxidation of the powder may be inhibited by coating the nickel and tin powder with a flux, for example zinc chloride. Also during selective laser sintering, the ambient temperature of the powder is preferably elevated, for example to a temperature on the order of 190°C. These conditions cause the tin to melt and infiltrate the powder particles of nickel, much as in the manner shown in FIGURE 11c described hereinabove. Upon cooling of the layer (beginning with movement of the laser beam away from the melted portion), the tin resolidifies, forming a solid mass of nickel powder particles within the resolidified tin. Successive layers are then similarly processed, until the desired part is fabricated. It should be noted that the melting of tin in a later layer serves to connect its part cross-section to that of the prior layer, so that a cohesive mass is formed in layerwise fashion.

After formation of the part, the mass has sufficient structural integrity to maintain its shape upon removal of the non-melted powder. The part is then annealed, for example at 850°C for eight hours, preferably in an inert atmosphere. During this anneal, nickel and tin atoms interdiffuse with one another, and chemically react to form the intermetallic compound Ni<sub>3</sub>Sn<sub>2</sub>. In the early stage of annealing at a temperature above the melting point of tin, remelting of the tin may cause a loss in the green strength of the part. Packing the part with a mold, formed of a material such as

alumina, will allow the part to maintain its shape during such annealing. Alternatively, the anneal may be done in stages, beginning with a low temperature (i.e., below the melting point of tin) anneal for sufficient time to begin the intermetallic formation, followed by a high temperature anneal to more rapidly complete the process.

Referring again to FIGURE 13, the melting point of this compound is on the order of 1260°C. It should be noted that the 10 high melting point temperature of the compound  $Ni_3Sn_2$  is practicably not attainable by irradiation of a laser in the context of selective laser sintering, much less a 25 watt  $CO_2$  laser as used in this method. As a result of this embodiment 15 of the invention, a low temperature process can form a part of a high melting point material. This method thus broadens the range of materials with which the selective laser sintering process may be used, to include refractory compounds such as the intermetallic compound of  $Ni_3Sn_2$ . Since parts formed of this material are able to withstand thermal and mechanical 20 conditions far greater than parts formed of polycarbonate, plastics, and other low temperature materials, these parts may be used in such applications as testing of the strength or functionality of an apparatus incorporating the part, and also as custom manufactured parts installed into actual end equipment.

FIGURE 14 is a phase diagram for another intermetallic system suitable for use with the method according to this embodiment of the invention is illustrated, namely the binary 30 system of nickel and aluminum. The desired binary intermetallic compound in this system is  $NiAl$ , which has a melting point of 1638°C. It should be noted that  $NiAl$  is an attractive material for such applications as aerospace, due to its ability to withstand extremely high temperatures while 35 maintaining increased strength and good resistance to oxidation

and corrosion. For this system, the powder mixture to be irradiated by the directed energy beam is preferably 50% aluminum and 50% nickel (atomic percent), and the directed energy beam must heat the powder in the selected regions to a temperature of at least 660°C to melt the aluminum. As in the prior examples, upon cooling, the molten aluminum will bind the solid nickel powder particles into a mass at the desired locations of the layer; subsequent layers are similarly processed to form the part. Post-processing high temperature annealing will form the intermetallic compound of NiAl, having a very high melting point. It should be noted that the melting point of NiAl is higher than that of either of the precursor elements.

The process described hereinabove may also be used to produce Ni<sub>3</sub>Al, which is another attractive aerospace compound in the nickel-aluminum system; the powder mixture for this intermetallic would include 25% aluminum and 75% nickel (atomic percent). In addition, a powder mixture including aluminum in the range from 25% to 50% can be used to produce a two phase material upon thermal annealing, namely Ni<sub>3</sub>Al-NiAl. For example, if the powder is a mixture of 35% aluminum and 65% nickel (atomic percent), the part produced according to this embodiment of the invention will be of the two phase material Ni<sub>3</sub>Al-NiAl, if equilibrium conditions prevail.

Other candidate binary systems for forming a high melting point intermetallic compound, and which are believed to be suitable for formation by way of lower temperature selective laser sintering as described hereinabove, include (without limitation thereto) Al-Nd, Al-Th, Al-Ti, Al-Y, Al-Yb, Al-Zr, Bi-La, Bi-Nd, Bi-Y, Bi-Zr, Ca-Si, Co-Nb, Co-W, Fe-Ti, Gd-Pb, Gd-Ru, Ge-Hf, In-Pd, In-Pr, Mn-Si, Nb-Sn, Pd-Sn, Pd-Ti, Pd-Tl, Pd-Zr, Pr-Sn, Si-Ti, Sn-Zr, Te-Zn, Th-Zn, and Y-Zn. Ceramic systems which have such characteristics as to be suitable for

this embodiment of the invention include  $\text{La}_2\text{O}_3\text{-B}_2\text{O}_3$ ,  $\text{Bi}_2\text{O}_3\text{-La}_2\text{O}_3$ ,  $\text{Bi}_2\text{O}_3\text{-Nb}_2\text{O}_5$ , and  $\text{V}_2\text{O}_5\text{-Al}_2\text{O}_3$ . It is contemplated that other binary systems, as well as tertiary, quaternary, and other even more complex compound systems of metals and ceramics (including 5 glass), will also be useful in the method according to this embodiment of the invention where a chemical reaction is enabled by exposure of precursor materials to the directed energy beam.

10 In addition, it is contemplated that the compound may be formed during the irradiation by the directed energy beam, such that a part can be formed of a material with different properties from the precursors without requiring subsequent annealing of the part. For a chemical reaction to occur, it is 15 of course necessary that the reaction kinetics be such that conversion of the precursor powders to the compound can occur in the time allotted for selective laser sintering. It should be noted that the time which the directed energy beam can remain over a particular location of the powder is probably 20 limited by the thermal conduction of the powder, as it is undesirable for non-irradiated portions of the powder adjacent to the irradiated beam to also undergo melting, sintering, or other thermal bonding, as such would compromise the resolution of the part being produced.

25

It should also be noted that, alternatively to the provision of a directed energy beam, other energy delivery techniques to selected portions of the powder layer may also be sufficient to cause the melting or reaction necessary to form 30 the part in the manner described hereinabove. For example, a mask may be provided in proximity to the powder, through which a light source is projected to selected portions of the powder receive the energy from the light source. Other techniques of delivering thermal energy to selected portions of the powder 35 surface will also be similarly applicable.

Turning to FIGURE 12, in yet another preferred embodiment a material 1201 is preferably deposited on surface 1200 and 5 second material 1203 is then deposited on material 1201 prior to sintering. Materials 1201 and 1203 preferably have different bonding or dissociation temperatures.

10 Operation

A fundamental concept of the present invention is the build up of a part in a layer-by-layer manner. That is, a part is considered a plurality of discrete cross-sectional regions 15 which cumulatively comprise the three-dimensional configuration of the part. Each discrete cross-sectional region has defined two-dimensional boundaries - of course, each region may have unique boundaries.

20 In the method, a first portion of powder 22 is deposited in the target area 26 and selectively sintered by the laser beam 64 to produce a first sintered layer 54 (FIGURE 2). The first sintered layer 54 corresponds to a first cross-sectional region of the desired part. The laser beam selectively sinters only 25 the deposited powder 22 within the confines of the defined boundaries.

There are, of course, alternative methods of selectively sintering the powder 22. One method is for the aim of the beam 30 to be directed in a "vector" fashion - that is, the beam would actually trace the outline and interior of each cross-sectional region of the desired part. Alternatively, the aim of the beam 64 is scanned in a repetitive pattern and the laser 12 modulated. In FIGURE 2, a raster scan pattern 66 is used and 35 is advantageous over the vector mode primarily in its

simplicity of implementation. Another possibility is to combine the vector and raster scan methods so that the desired boundaries of the layer are traced in a vector mode and the interior irradiated in a raster scan mode. There are, of course, trade-offs associated with the method chosen. For example, the raster mode has a disadvantage when compared to the vector mode in that arcs and lines which are not parallel to the axes 68, 70 of the raster pattern 66 of the laser beam 64 are only approximated. Thus, in some cases resolution of the part can be degraded when produced in the raster pattern mode. However, the raster mode is advantageous over the vector mode in the simplicity of implementation.

Turning to FIGURE 1, the aim of the laser beam 64 is scanned in the target area 26 in a continuous raster pattern. Broadly speaking, the driver 50 controls galvanometers 48, 49 to make the raster pattern 66 (see FIGURE 2). Shifting movement of the mirror 46 controls movement of the aim of the laser beam 64 in the fast scan axis 68 (FIGURE 2), while movement of the mirror 47 controls movement of the aim of the laser beam 64 in the slow scan access 70.

The present position of the aim of the beam 64 is fed back through the driver 50 to the computer 40 (see FIGURE 3). As described below, in more detail, the computer 40 possesses information relating to the desired cross-sectional region of the part then being produced. That is, a portion of loose powder 22 is dispensed into the target area 26 and the aim of the laser beam 64 moved in its continuous raster pattern. The computer 40 modulates the laser 12 to selectively produce a laser beam at desired intervals in the raster pattern 66. In this fashion, the directed beam of the laser 12 selectively sinters the powder 22 in the target area 26 to produce the desired sintered layer with the defined boundaries of the desired cross-sectional region. This process is repeated

layer-by-layer with the individual layers sintered together to produce a cohesive part - e.g. part 52 of FIGURE 2.

In operation, the wavelength of laser 12 may be varied to  
5 produce higher absorptivity of energy by selected materials in the powder relative to other materials in powder 22. In operation, blended, coated or other combinations of powders are preferably selected to produce sintered product with characteristics including but not limited to, close dimensional  
10 tolerances, structural integrity and required mechanical behavior.

#### Interface and Software

15 The interface hardware operatively interconnects the computer 40 with the laser 12 and galvanometers 47, 48. The output port of the computer 40 (see FIGURES 1 and 3) is directly connected to the laser 12 to selectively modulate the  
20 laser 12. When operated in the pulsed mode, the laser 12 is easily controlled by digital inputs to the pulsed gate input of the laser. Galvanometer 48 is driven by the function generator driver 50 to drive the beam in the fast scan axis 68 independent of any control signals from the computer 40.  
25 However, a position feedback signal from the galvanometer 48 is fed to a voltage comparator 74 as shown in FIGURE 3. The other input to the comparator is connected to the digital-to-analog convertor 76 which is indicative of the least significant six bits (bits 0-5) of the user port of the computer 40. As shown  
30 in FIGURE 3, the output of the voltage comparator 74 is connected to the flag line on the user port of the computer 40. When the voltage comparator determines that the feedback signal from the galvanometer 48 crosses the signal from the digital-to-analog convertor 76, the flag line goes low causing a  
35 nonmaskable interrupt. As discussed below, the nonmaskable

interrupt causes the next byte of data to put out on the user port of a computer 40.

Finally, as shown in FIGURE 3, the galvanometer 49 driving 5 the aim of the laser beam 64 in the slow scan axis 70, is controlled by a second digital to analog convertor 78. The digital-to-analog convertor 78 is driven by a counter 79 which increments with each sweep of the aim of the beam 64 in the fast scan axis 68. The eight byte counter is designed to 10 overflow after 256 scans in the fast scan axis 68 to start a new cycle or raster scan pattern 66.

Preferably, the control information (i.e. defined boundaries of the cross-sectional regions) data for each raster 15 pattern 66 would be determined by a CAD system given the overall dimensions and configuration of the part to be produced. Whether programmed or derived, the control information data for each raster pattern 66 is stored in the computer memory as a series of eight bit words. The data 20 format represents a pattern of "on" and "off" regions of the laser 12, versus distance along the raster pattern 66 traveled by the aim of the beam 64. The data is stored in a "toggle-point" format where the data represents the distance along each raster scan pattern 66 where the laser is modulated (i.e. 25 turned from on to off or from off to on). Although a "bit map" format might be used, the toggle point format has been found more efficient for the production of high resolution parts.

For each eight bit word, the least significant six bits 30 (bits 0-5) represent the location of the next toggle point - i.e. the next location for modulation of the laser 12. The next bit (bit 6) represents whether the laser is on or off immediately before the toggle point identified in the least significant six bits. The most significant bit (MSB or bit 7) 35 is used for looping and for controlling the slow scan axis 70

of the aim of the beam 64. Because the Commodore 64 had limited memory, looping was required - it being understood that a computer 40 with more memory would not require looping.

5 FIGURE 6 represents the flow chart for the data metering program. The data metering program is run whenever the flagline goes low causing a non-maskable interrupt (see FIGURE 3). The interrupt causes the microprocessor of the computer 40 to retrieve a two byte interrupt vector which points to the  
10 location in memory where program control is transferred at interrupt. As shown in FIGURE 6, the data metering program first pushes the registers onto the stack and then loads the next byte of data into the accumulator. The data word is also output to the user port with the sixth bit used to modulate the  
15 laser 12 (FIGURE 3).

As shown in FIGURE 6, the most significant bit (MSB or bit 7) of the data word in the accumulator is examined. If the value of the most significant bit is one, that means the end of  
20 the loop has not been reached; therefore the data pointer is incremented, registers are restored from the stack, and the data metering program is exited, returning control to the microprocessor at the location of interrupt. If the most significant bit in the accumulator is zero, the data word is  
25 the last word in the loop. If the data word is the last word in the loop, the next bit in memory is a loop counter and the following two bytes are a vector pointing to the top of the loop. As can be seen from FIGURE 6, if the most significant bit equals zero (end of the loop) the loop counter (next bit)  
30 is decremented and analyzed. If the loop counter is still greater than zero, the data pointer assumes the value from the next two memory bytes after the loop counter, registers are pulled from the stack and program control returns to the location of interrupt. On the other hand, if loop counter is  
35 zero, the data pointer is incremented by three and the loop

counter is reset to ten before exiting the program. It can be appreciated that the need for such looping is absolved if the memory size of the computer 40 is adequate.

5    Example

In FIGURES 4 and 5, an example part 52 is illustrated. As can be seen from the drawing, the example part 52 assumes an unusual shape in that it is not symmetrical and would be 10 difficult to fabricate using conventional machining methods. For reference purposes, the part 52 includes an outer base structure 80 having an interior cavity 82 and a pillar 84 disposed within the cavity 82 (see FIGURE 4). FIGURE 5 shows the part 52 within the confinement structure 28 defining the 15 target area 26 illustrated in FIGURE 1. As shown in FIGURE 5, some of the powder 22 is loose, while the remainder of the powder is selectively sintered to comprise the structure of the part 52. FIGURE 5 is shown in vertical section with parts broken away and outlined in phantom to show the sintered 20 cohesive portions of the part 52.

FIGURE 7 shows a horizontal cross-sectional region, taken along line 7-7 of FIGURE 4. FIGURE 7 represents a discrete layer 86 associated with the cross-sectional region of the part 25 being produced. As such, the sintered layer 86 of FIGURE 7 is a product of a single raster pattern 66 as illustrated in FIGURE 2.

For reference purposes, a sweep line through the sintered 30 layer 86 has been labeled "L." FIGURE 8 illustrates the software and hardware interface operation during the sweep L. The top graph shows the position of feedback signal from the fast axis galva 48 and the output signal of the first digital to analog convertor 76 (compare FIGURE 3). The voltage 35 comparator 74 generates an output signal to the flag line of

the computer 40 every time the feedback signal and first D/A output signal cross.

In the top graph of FIGURE 8, these points are labeled T to  
5 represent toggle points. As can be seen from the bottom graph  
of FIGURE 8, the flag line generates a nonmaskable interrupt  
corresponding to each toggle point T. The sixth bit of each  
data word is analyzed and the current state of the laser 12  
will reflect the value. The penultimate graph of FIGURE 8  
10 shows the laser modulation signal for the sweep line L of  
FIGURE 7. The second graph of FIGURE 8 shows that a high-going  
edge in the most significant bit will be encountered at the end  
of each sweep of the aim of the laser beam 64 in the fast scan  
axis 68. As shown in FIGURES 3 and 6, the counter 79  
15 increments on a high going edge, and outputs a signal to the  
second digital-analog convertor 78 to drive the slow axis  
galvanometer 49.

As can be seen from the example illustrated in the drawing,  
20 parts of complex shape can be produced with relative ease.  
Those skilled in the art will appreciate that the part 52  
illustrated in FIGURE 4 would be difficult to produce using  
conventional machining methods. In particular, machine tool  
access would make the fabrication of cavity 82 and pillar 84  
25 difficult, if not impossible, to produce if the part 52 were of  
a relatively small size.

In addition to avoiding the access problem, it will be  
appreciated that the production accuracy is not dependent upon  
30 machine tool wear and the accuracy of mechanical components  
found in conventional machine tools. That is, the accuracy and  
tolerances of the parts produced by the method and apparatus of  
the present invention are primarily a function of the quality  
of the electronics, the optics, and the implementing software.  
35 Of course, heat transfer and material considerations do affect

the tolerances obtainable.

Those skilled in the art will appreciate that conventional machining techniques require considerable human intervention and judgment. For example, a conventional machining process, such as milling, would require creativity to make such decisions as tool selection, part segmenting, sequence of cuts, etc. Such decisions would even be more important when producing a control tape for a tape control milling machine.

On the other hand, the apparatus of the present invention only requires the data relating to each cross-sectional region of the part being produced. While such data can be simply programmed into the computer 40, preferably, the computer 40 includes a CAD/CAM system. That is, the CAD/CAM portion of the computer 40 is given the overall dimensions and configurations of the desired part to be produced and the computer 40 determines the boundaries for each discrete cross-sectional region of the part. Thus, a vast inventory of part information can be stored and fed to the computer 40 on a selectable basis.

The apparatus 10 produces a selected part without set-up time, part specific tooling, or human intervention. Even the complex and expensive dies associated with powder metallurgy and conventional casting techniques are avoided.

While large quantity production runs and certain part material characteristics might be most advantageously made using conventional fabrication techniques, the method and apparatus 10 of the present invention is useful in many contexts. In particular, prototype models and casting patterns are easily and inexpensively produced. For example, casting patterns are easily made for use in sand casting, lost wax casting, or other forming techniques. Further, where desired quantities are very small, such as with obsolete replacement parts, production of such replacement parts using the apparatus 10 of the present invention has many advantages. Finally, the

use of the apparatus 10 may be useful where size of production facilities is a major constraint, such as on-ship or in outer space.

5        Further modification and alternative embodiments of the apparatus of this invention will be apparent to those skilled in the art in view of this description. Accordingly this description is to be construed as illustrative only and is for the purpose of teaching those skilled in the art the manner of  
10 carrying out the invention. It is to be understood that the forms of the invention herein shown and described are to be taken as the presently preferred embodiments. Various changes may be made in the shape, size and arrangement of parts. For example, elements or materials may be substituted for those  
15 illustrated and described herein, parts may be reversed, and certain features of the invention may be utilized independently of the use of other features, all as would be apparent to one skilled in the art after having the benefit of this description of the invention.

## WE CLAIM:

1. A method of producing a part, comprising:  
depositing a powder onto a target surface, said powder  
comprising first and second materials;  
heating a selected portion of said powder to define a cross-sectional region of the part; and  
reacting the first and second materials in said cross-sectional region of the part to form a chemical compound of  
said first and second materials.
2. The method of claim 1, wherein said heating step heats said powder to a temperature between the melting point of said first material and the melting point of said second material.
3. The method of claim 2, wherein the melting point of said chemical compound is higher than the melting point of said first material.
4. The method of claim 3, wherein the melting point of said chemical compound is higher than the temperature to which the selected portion of the powder is heated in said heating step.
5. The method of claim 1, further comprising:  
depositing a second layer of said powder after said heating step; and  
heating a selected portion of said second layer of powder to define a second cross-sectional region of the part.
6. The method of claim 5, wherein said reacting step comprises:  
removing portions of the powder not within the defined cross-section of the part, after said step of heating said second layer of powder; and

heating the part after said removing step.

7. The method of claim 1, wherein said reacting step is performed during said heating step.

5

8. The method of claim 1, wherein said heating step comprises:

directing an energy beam at selected portions of said powder.

10

9. The method of claim 8, wherein the directing step comprises:

directing a laser beam at selected portions of said powder.

15

10. The method of claim 1, wherein said first material comprises tin.

20

11. The method of claim 10, wherein said second material comprises nickel.

12. The method of claim 1, wherein said second material comprises nickel.

25

13. The method of claim 12, wherein said first material comprises aluminum.

14. The method of claim 1, wherein said first and second materials are metals.

30

15. The method of claim 1, wherein said first and second materials are ceramics.

35

16. The method of claim 1, wherein the chemical compound formed by said reacting step is a stable compound.

17. The method of claim 1, wherein the chemical compound formed by said reacting step is a metastable compound.

5        18. A part produced by the method comprising the steps of: depositing a powder onto a target surface, said powder comprising first and second materials;

heating a selected portion of said powder to define a cross-sectional region of the part; and

10        reacting the first and second materials in said cross-sectional region of the part to form a chemical compound of said first and second materials.

15        19. The part of claim 18, wherein said heating step heats said powder to a temperature between the melting point of said first material and the melting point of said second material; and wherein the melting point of said chemical compound is higher than the temperature to which the selected portion of the powder is heated in said heating step.

20        20. The part of claim 18, wherein the method further comprises:

depositing a second layer of said powder after said heating step; and

25        heating a selected portion of said second layer of powder to define a second cross-sectional region of the part;

and wherein said reacting step comprises:

removing portions of the powder not within the defined cross-section of the part, after said step of heating said 30 second layer of powder; and

heating the part after said removing step.

21. The part of claim 18, wherein said reacting step is performed during said heating step.

22. The part of claim 18, wherein said first and second materials are metals.

23. The part of claim 22, wherein said compound is an intermetallic compound having a melting point higher than the melting point of said first material.

24. The part of claim 18, wherein said first and second materials are ceramics.



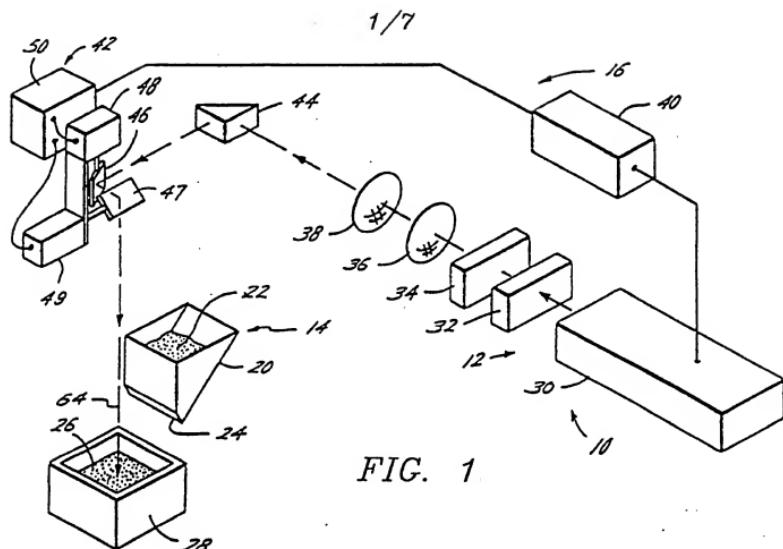
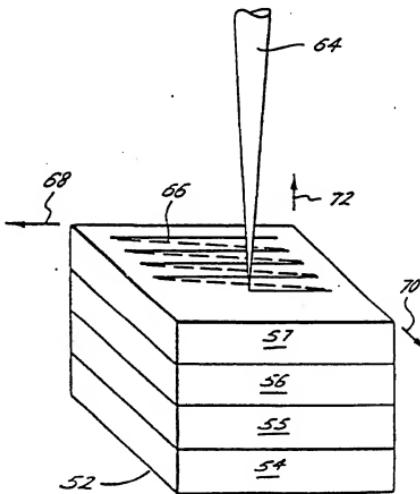


FIG. 1

FIG. 2



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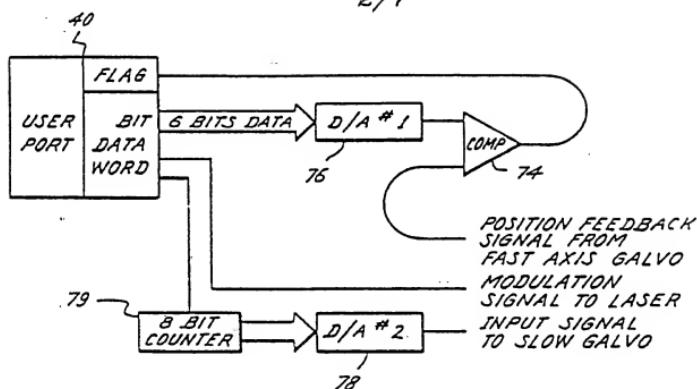


FIG. 3

FIG. 4

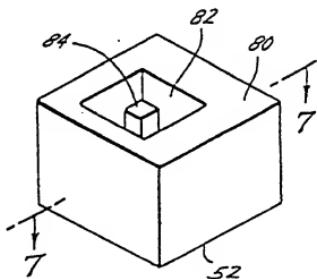
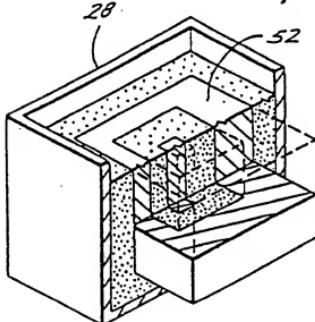
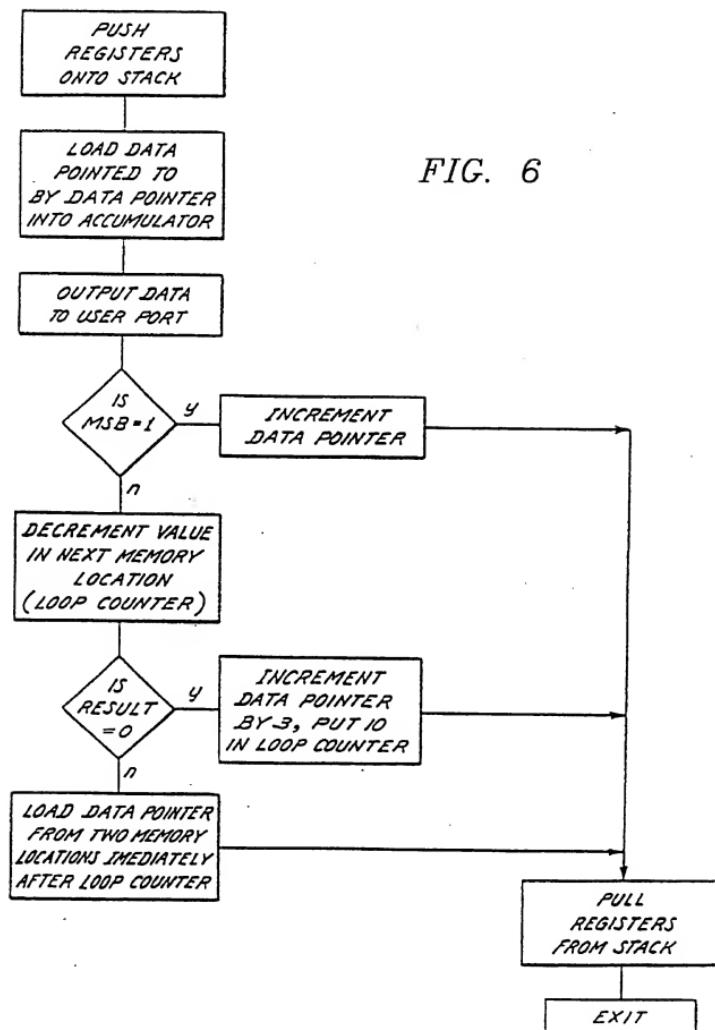


FIG. 5



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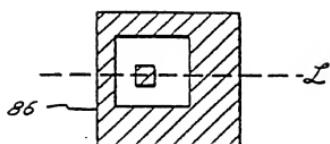
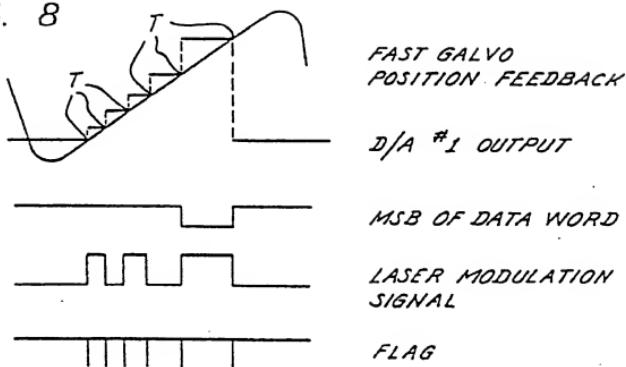


FIG. 7

FIG. 8



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FIG. 9

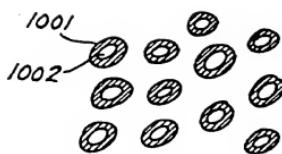


FIG. 10

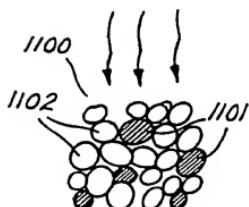


FIG. 11a

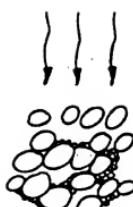


FIG. 11b

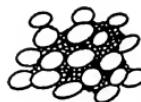


FIG. 11c

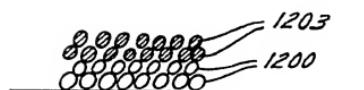


FIG. 12



FIG. 11d

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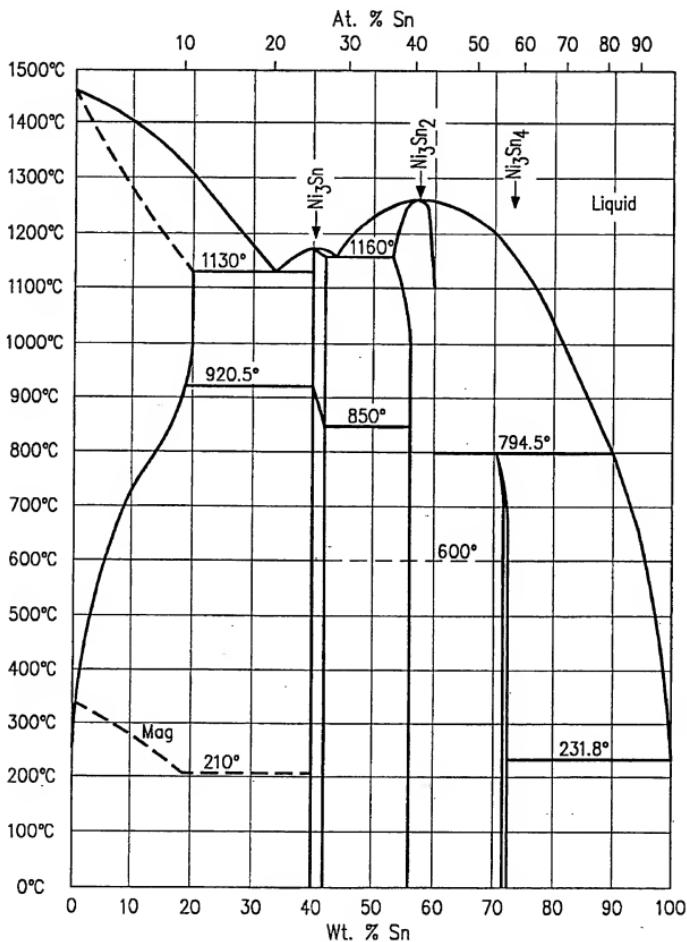


FIG. 13

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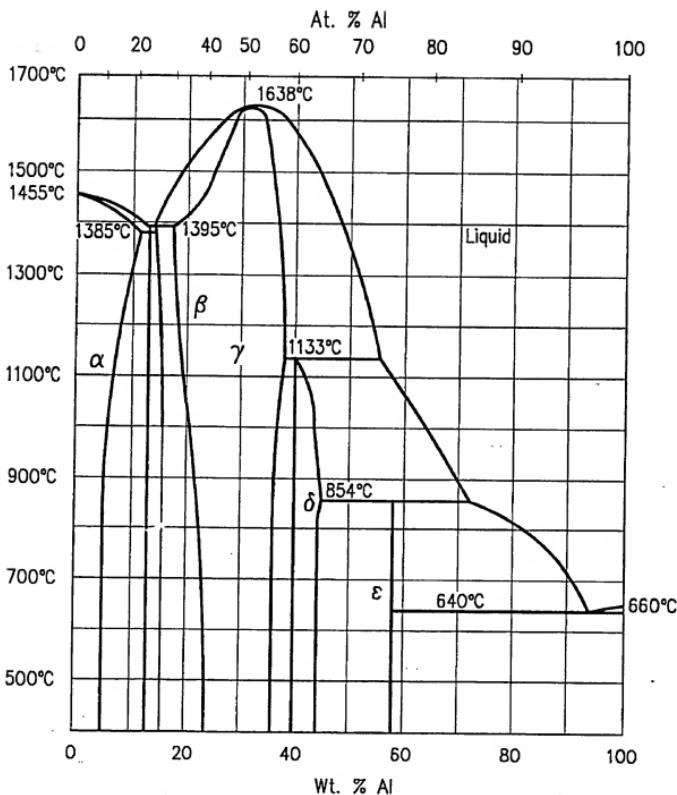


FIG. 14

## INTERNATIONAL SEARCH REPORT

International Application No. PCT/US91/09313

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all)<sup>3</sup>

According to International Patent Classification (IPC) or to both National Classification and IPC

IPC. CL(5): B27N 3/00; B32B 31/00; B23K 9/00; B29C 67/00

US CL : 156/062.2, 272.8; 219/121.66, 121.8; 264/058, 113,125; 428/206, 402.2, 404,406,407; 425/174; 419/005,008

## II. FIELDS SEARCHED

Minimum Documentation Searched<sup>4</sup>

Classification System | Classification Symbols

U.S.

156/062.2,089,272.8; 219/121.66,121.8,121.85; 264/058,113,125  
428/206,357,361,402,402.2,404,406,407; 425/174; 419/005,008Documentation Searched other than Minimum Documentation  
to the Extent that such Documents are Included in the Fields Searched<sup>5</sup>III. DOCUMENTS CONSIDERED TO BE RELEVANT<sup>14</sup>

Category <sup>6</sup>	Citation of Document, <sup>15</sup> with indication, where appropriate, of the relevant passages <sup>17</sup>	Relevant to Claim No. <sup>18</sup>
A	US,A 4,752,352 (PEYGIN) 21 June 1988	1-24

<sup>3</sup> Special categories of cited documents:<sup>13</sup><sup>"A"</sup> document defining the general state of the art which is not considered to be of particular relevance<sup>"E"</sup> earlier document but published on or after the international filing date<sup>"L"</sup> document which may throw doubt on priority claim(s) or which is cited to establish the publication date of another document under special reasons (as specified)<sup>"O"</sup> document referring to an oral disclosure, use, exhibition or other means<sup>"P"</sup> document published prior to the international filing date but later than the priority date claimed<sup>"T"</sup> later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention<sup>"X"</sup> document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step<sup>"Y"</sup> document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.<sup>"A"</sup> document member of the same patent family.

## IV. CERTIFICATION

Date of the Actual Completion of the International Search<sup>19</sup>

02 April 1992

Date of Mailing of this International Search Report<sup>19</sup>

16 APR 1992

International Searching Authority<sup>20</sup>

ISA/US

Signature of Authorized Officer<sup>21</sup>

Wm. A. Powell